

PATENT SPECIFICATION

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(54) PAPER PRODUCT INCORPORATING FIBROUS POLYESTER MATERIAL

(71) We, EASTMAN KODAK COMPANY, a Company organised under the Laws of the State of New Jersey, United States of America, of 343 State Street, Rochester, New York 5 14650, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to a paper product incorporating a water-dispersible fibrous polyester material.

15 Synthetic fibre reinforcement of paper products has long been of interest to paper manufacturers. For example, specialty papers including synthetic fibres have much improved tear resistance, bursting and wet strength as well as better fold characteristics. The incorporation 20 of such fibres further improves the dimensional stability, resistance to degradation and electrical properties of paper products. These superior paper properties, however, will not be achieved unless each fibre is well wetted-out 25 and free of adherent air bubbles which would cause flotation, flocculation or agglomeration.

30 Numerous synthetic fibres have been employed both experimentally and commercially by the paper industry. Each fibre type differs substantially from the others with respect to the problems encountered when they are employed in the paper making process, the problems including the extent of fibre dispersibility in water, bonding between fibres in the paper, 35 and the fibrillation of the fibres in the paper making machinery.

40 Polyester fibres have been employed by paper manufacturers generally in staple form. These staple fibres are hydrophobic, in common with most synthetic fibres, and will not disperse readily in water. Wetting agents have been employed in efforts to disperse the fibres more uniformly in the paper making slurry. Use of these agents in the quantities necessary

45 to assist in fibre dispersion also creates large quantities of foam, which is highly undesirable and in most instances cannot be tolerated by the paper manufacturer. Several fibre surface treatments have also been employed in efforts to achieve a satisfactory degree of dispersion. This has been accomplished with moderate success, but the present invention provides significantly improved dispersibility.

50 In accordance with the present invention, there is provided a reinforced paper product containing up to 25% by weight of hydrophilic water-dispersible polyester fibres, the surfaces of which have been substantially hydrolyzed.

55 The polyesters with which the invention is primarily concerned are poly(ethylene terephthalate); and poly(1,4-cyclohexylenedimethylene terephthalate). Subsequent to spinning, the fibre materials manufactured from these polymers are generally drawn, crimped and heat-set. These treatments improve the physical characteristics of the material and condition it for textile usage. Of course, additional treatments or processing techniques may be employed depending upon the particular polyester and the manner in which it is to be employed. For example, synthetic fibres that are intended for inclusion in paper are not crimped and are generally supplied to the paper manufacturer in staple form.

60 Treatment of the polyester fibre surfaces, preferably with dilute alkali solution, to achieve substantial saponification or hydrolysis, materially improves their dispersibility. Such polyester fibres, because of their modified surfaces, become hydrophilic and thus can be more easily and uniformly dispersed in water without the aid of wetting agents. Further, dilute alkali treatment may be employed by either the fibre producer or the paper manufacturer, whichever may find it more convenient, and the fibre manufacturer may hydrolyse the polyester fibre surfaces without extensive process modifica- 70 75 80 85

tions. Therefore, the invention offers flexibility in processing, a feature not previously afforded either the fibre producer or the paper manufacturer.

5 Commercial polyester fibre production techniques usually include provisions for the storage of the continuous spun filaments in can-like containers immediately subsequent to the spinning operation. Filament ends are withdrawn from a plurality of these containers and combined into a continuous tow. This tow is then drafted sequentially in a water bath and steam. If desired, the water bath employed for drafting may also be used as a convenient 10 medium within which to surface treat the polyester filaments. Therefore, it is only necessary to prepare the water bath, incorporating that amount of sodium hydroxide or similar substance that will produce the solution type and 15 concentration desired.

10 After drafting, the polyester fibrous material is normally heat-set and then cut into staple lengths of between 3/16-inch and 5/8-inch. If, however, the filaments have not been 20 surface treated during the drafting operation, they can be immersed in a dilute alkali solution subsequent to the heat-setting and prior to the cutting operations. This is only one of several 25 alternative arrangements available to the fibre producers. In another arrangement, for example, the fibrous material may be converted to staple form prior to heat-setting and the sodium hydroxide treatment may be performed at virtually any stage of the processing, either 30 before or after heat-setting. It should also be noted that the fibrous material may be alkali treated in any one of a variety of ways, for example by spraying or roll application. Immersion, however, appears to be the most 35 practical, primarily because the usual polyester fibrous material manufacturing techniques include immersion steps for other specific reasons, and it is therefore convenient to accomplish this surface treatment in a like manner.

40 The alkaline treatment described substantially hydrolyses or saponifies the polyester fibre surfaces, generating hydroxyl ($-\text{OH}$), carboxyl ($-\text{COOH}$), and / or salt ($-\text{COO}^-\text{Na}^+$) groups that in effect render the 45 fibres hydrophilic. Although it is possible to generate such groups by hydrolysis with other acid or alkaline reagents, sodium hydroxide is preferred. In practice the specific conditions of 50 treatment may vary. For example, the time of treatment may change depending upon the alkali concentration, the solution temperature, and the degree of saponification desired. Solution 55 concentrations of between 0.1% and 1.0%

by weight and solution temperatures between the ambient and 70°C. are preferred.

60 Preferably, the denier per filament of the fibres is from 1 to 4.

65 As has been stressed, the dispersion of synthetic fibres in water is of particular importance to the paper manufacturer. If, during the paper making operation, such fibres begin to agglomerate or flocculate, the paper quality will be significantly affected.

70 Polyester fibres treated in the manner described produce surprising and unexpected results when their dispersibility is evaluated, as can be seen from the following examples:

EXAMPLE 1.

75 1.5 denier uncrimped 1/4" fibres of poly (ethylene terephthalate) were placed in a graduated cylinder containing a 0.1% by weight room temperature sodium hydroxide/water solution until a 0.5% by weight fibre/liquid consistency was reached. The cylinder was shaken until the fibres were evenly dispersed throughout the solution. After agitation the cylinder was allowed to stand motionless and the degree of fibre settling was observed after 30 seconds. No settling or agglomeration was observed.

EXAMPLE 2.

80 Essentially the same procedure as in Example 1 was followed, except that the graduated cylinder contained only pure water. In this instance, after a 30 second settling period, considerable agglomeration occurred.

EXAMPLE 3.

85 The fibre and water mixtures of Examples 1 and 2 were diluted to a 0.1% consistency (i.e. 0.1% by weight of poly(ethylene terephthalate) fibre in the liquid), there being no addition of sodium hydroxide to the solution to either cylinder. Again the cylinders were shaken and allowed to stand. After approximately 30 seconds, it was apparent that the sodium hydroxide treated fibres continued to demonstrate the improvement in dispersibility.

EXAMPLE 4.

90 A poly(ethylene terephthalate) tow band consisting of approximately 42,800 filaments (6 D/F-spun denier) was drafted 3:1 (first stage) in a bath containing a 0.25% by weight sodium hydroxide solution at about 68°C. The tow was further drafted at a ratio of about 1.34:1 in a steam chest heated to approximately 150°C. thereby giving an overall draft of about 4:1. The drafted tow (approximately 60,000 final denier) was staple cut and heat-set for about 6.3 seconds. Subsequent to this treatment the physical properties of the fibre were:

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120	Fibre Length	1/4 inch (nominal)
	% NaOH on Fibre	0.08%
	Denier/Filament	(D/F) — 1.2
	Tenacity	6/3 grams/denier (i.e. 6 grams for each 3 denier fibre)
	% Elongation	19.0%

EXAMPLE 5.

Uncrimped polyester fibres were dispersed in a beaten slurry (30 sec. Williams slowness) of Alberta Hi-brite Bleached Kraft paper stock 10
 5 at a weight ratio of 25% polyester to 75% paper stock. In one case the polyester fibre was treated with a 0.1% by weight sodium

hydroxide solution at 60°C. for 15 minutes. In another case the polyester was added to the paper stack in an untreated form. The treated polyester fibres became wetted much more easily and therefore dispersed more readily and evenly.

	100% Alberta Paper Stock	75% Paper Stock 25% Polyester (treated)	75% Paper Stock 25% Polyester (untreated)
Weight (lb/1000 sq. ft.)	10-1/2	10-1/2	9-1/2
Elmendorf Tear	44	192	159
Perkins Mullen (psi)	47	27	25
Wet Tensile (Kg/15 mm)	0.7	0.5	0.5
Valley Carbonate Pen.	1	1	1
2 min. Wet Expansion (%)	1.1	0.2	0.15
M.I.T. Folds	598	2855	857
Edge Tear	0.748	2.530	2.512

15 WHAT WE CLAIM IS:—

1. A reinforced paper product containing up to 25% by weight of hydrophilic water-dispersible polyester fibres, the surfaces of which have been substantially hydrolysed. 35
 20 2. A reinforced paper product as claimed in Claim 1, wherein the polyester fibres are uniformly dispersed through the paper.
 3. A reinforced paper product as claimed in Claim 1 or 2, wherein the surfaces of the fibres have been hydrolysed by treatment with dilute alkali solution. 40
 25 4. A reinforced paper product as claimed in Claim 2, wherein the surfaces of the fibres have been hydrolysed by immersion in dilute sodium hydroxide solution.
 30 5. A reinforced paper product as claimed in any one of Claims 1 to 4, wherein the fibres

are in staple form and are between 3/16" and 5/8" in length.

6. A reinforced paper product as claimed in any one of Claims 1 to 5, wherein the denier per filament of the fibres is from 1 to 4.

7. A reinforced paper product as claimed in any one of Claims 1 to 6, wherein the polyester is poly(ethylene terephthalate).

8. A reinforced paper product as claimed in any one of Claims 1 to 6, wherein the polyester is poly(1,4 - cyclohexylenedimethylene terephthalate).

9. A reinforced paper product as claimed in Claim 1 and substantially as hereinbefore described. 45

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